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(54) HIGH-RESISTIVITY RARE EARTH MAGNET AND ITS MANUFACTURING METHOD

(57)Abstract:

PROBLEM TO BE SOLVED: To provide a sintered magnet which has a high coercive force, is high enough in resistivity to restrain generation of an eddy current even if it is used for a motor where it is exposed to a fluctuating magnetic field, and manufactured at a low cost.

SOLUTION: This high resistivity rare earth magnet is formed of material that contains one or more rare earth oxides R' mOn (wherein, R' is one or more rare earth elements including Y; (m) and (n) are 1, 2 or 2, 3 when R' is Ce, 2, 3 or 6, 11 when R' is Pr, 2, 3 or 4, 7 when R' is Tb, and 2, 3 when R' is other rare earth elements) and/or rare earth element R'' (R'' is one or more earth elements, including Y), a B composite oxide, and the residue which is R-T-B alloy (wherein, R is one or more rare earth elements including Y; T is Fe and/or Co) and/or R-T-M-B alloy (wherein, R is one or more earth elements including Y; T is Fe and/or Co; M is one or more elements selected out of Al, Si, Ti, V, Cr, Cu, Zr, Nb, Mo, Ta, W, Ga, and Sn).

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(54) 【発明の名称】 高比電気抵抗性希土類磁石及びその製造方法

(57) 【要約】

【解決手段】 1種以上の希土類酸化物 $R' m O n$

(R' は Y を含む希土類元素の1種以上で、 m 、 n は、それぞれ Ce の場合 1, 2 又は 2, 3、Pr の場合 2, 3 又は 6, 11、Tb の場合 2, 3 又は 4, 7、その他は全て 2, 3 である) 及び/又は希土類元素 R''

(R'' は Y を含む希土類元素の1種以上) と B との複合酸化物を含み、残部が R-T-B 合金 (R は Y を含む希土類元素の1種以上、T は Fe 及び/又は Co) 及び/又は R-T-M-B 合金 (R は Y を含む希土類元素の1種以上、T は Fe 及び/又は Co、M は Al, Si, Ti, V, Cr, Cu, Zr, Nb, Mo, Ta, W, Ga, Sn のいずれかより選ばれた1又は2以上の元素) であることを特徴とする高比電気抵抗性希土類磁石。

【効果】 本発明によれば、高い保磁力及びモータ等変動する磁界にさらされるような使用条件でも渦電流の発生が抑えられる大きな比電気抵抗を持つ焼結磁石を、低コストで製造できる。

【特許請求の範囲】

【請求項 1】 1 種以上の希土類酸化物 $R'mOn$

(R' は Y を含む希土類元素の 1 種以上で、m、n は、それぞれ Ce の場合 1、2 又は 2、3、Pr の場合 2、3 又は 6、11、Tb の場合 2、3 又は 4、7、その他は全て 2、3 である) 及び/又は希土類元素 R''

(R'' は Y を含む希土類元素の 1 種以上) と B との複合酸化物を含み、残部が R-T-B 合金 (R は Y を含む希土類元素の 1 種以上、T は Fe 及び/又は Co) 及び/又は R-T-M-B 合金 (R は Y を含む希土類元素の 1 種以上、T は Fe 及び/又は Co、M は Al、Si、Ti、V、Cr、Cu、Zr、Nb、Mo、Ta、W、Ga、Sn のいずれかより選ばれた 1 又は 2 以上の元素) であることを特徴とする高比電気抵抗性希土類磁石。

【請求項 2】 R-T-B 及び/又は R-T-M-B 合金が、Nd₂Fe₁₄B 型の結晶構造を持つ相を有することを特徴とする請求項 1 記載の高比電気抵抗性希土類磁石。

【請求項 3】 希土類酸化物 $R'mOn$ 及び/又は R'' と B との複合酸化物の含有量が 0.0005~40 重量%である請求項 1 又は 2 記載の高比電気抵抗性希土類磁石。

【請求項 4】 R-T-B 合金 (R は Y を含む希土類元素の 1 種以上、T は Fe 及び/又は Co) 及び/又は R-T-M-B 合金 (R は Y を含む希土類元素の 1 種以上、T は Fe 及び/又は Co、M は Al、Si、Ti、V、Cr、Cu、Zr、Nb、Mo、Ta、W、Ga、Sn のいずれかより選ばれた 1 又は 2 以上の元素) のインゴット合金又は急冷薄帯を粉砕する工程において、B₂O₃ 粉末を混合、粉砕し、磁場中配向成形後、焼結することを特徴とする高比電気抵抗性希土類磁石の製造方法。

【請求項 5】 R-T-B 合金が、R が 8~40 原子%、B が 0.1~10 原子%、残部が T である請求項 4 記載の高比電気抵抗性希土類磁石の製造方法。

【請求項 6】 B₂O₃ 粉末の混合量を 0.0001~15 重量%とする請求項 4 又は 5 記載の高比電気抵抗性希土類磁石の製造方法。

【請求項 7】 B₂O₃ 粉末の平均粒径が 0.1 μm~5 mm であることを特徴とする請求項 4 乃至 6 のいずれか 1 項記載の高比電気抵抗性希土類磁石の製造方法。

【請求項 8】 請求項 4 乃至 7 のいずれか 1 項記載の方法により製造した高比電気抵抗性希土類磁石を熱処理することを特徴とする高比電気抵抗性希土類磁石の製造方法。

【発明の詳細な説明】

【0001】

【発明の属する技術分野】 本発明は、特に、回転機器、電子部品、電気機器の産業分野で有用な、高比電気抵抗

性希土類磁石及びその製造方法に関するものである。

【0002】

【従来の技術】 R (R は Y を含む希土類元素の 1 種以上、以下同じ)、Fe、Co、B からなる永久磁石、特に R として Nd を主成分とする希土類磁石は、その磁気特性の高さから電子・電気機器産業の分野において、広く利用されている。

【0003】 例えば、永久磁石式回転機器には、従来安価なフェライト磁石が主に使用されてきた。しかし近年、回転機器の更なる小型化、効率化の要求に対して、高価ではあるが磁気特性の高い希土類磁石が使用されるようになった。一般に市販されている希土類磁石のうち、Sm-Co 系磁石はキュリー温度が高いため、磁気特性の温度変化が小さい。また耐食性も高く、表面処理を必要としない。しかし、原料として Co を使用しているため、非常に高価である。一方、Nd-Fe-B 系磁石は永久磁石の中で飽和磁化が最も高く、また原料で Co を使用しないので安価である。しかし、キュリー温度が低いため、磁気特性の温度変化が大きく、耐熱性に劣る。同時に耐食性も劣っているため、用途によっては適当な表面処理を必要とする。

【0004】

【発明が解決しようとする課題】 希土類磁石は金属であるので、比電気抵抗は酸化物であるフェライトの比電気抵抗の 100 分の 1 程度の 100~200 μΩcm 程度と小さい。従って、モータなど回転機器でこの希土類磁石が変動する磁界にさらされながら使用される場合、電磁誘導により発生した渦電流が大量に流れ、その電流によるジュール熱により永久磁石が発熱する。永久磁石の温度が高くなると、特に Nd-Fe-B 系磁石は磁気特性の温度変化が大きいため、磁気特性が低下し、その結果モータの効率も落ちる。

【0005】 この対策の一つとして、磁石の保磁力を大きくすることが挙げられる。磁石の保磁力を大きくすることにより、磁石の耐熱性が増し、不可逆減磁を起こしにくくなる。特に Nd-Fe-B 系磁石はそのキュリー温度の低さから磁気特性の温度変化が大きく、その保磁力を高める様々な試みがなされてきた。

【0006】 その保磁力を高めるのに最も効果が現れた方法は、その主相となる R₂ (Fe、Co)₁₄B 相の R の一部を Dy や Tb といった重希土類に置換し、結晶磁気異方性を高めるやり方であった。しかしこの方法では磁石の飽和磁化は小さくなり、また重希土類は非常に高価であるため磁石のコストがあがる。

【0007】 渦電流を抑えるための方法に、永久磁石を小分割して絶縁する方法がある。この方法は、磁石の分割数に反比例して発熱量を小さくする効果があるが、磁石加工のコストを上げてしまう。

【0008】 そこで、渦電流に対する抜本的な対策として、希土類磁石そのものの比電気抵抗を大きくしようと

する試みがなされている。

【0009】樹脂バインダを使用したNd-Fe-B系希土類ボンド磁石の比電気抵抗は、Nd-Fe-B系焼結磁石と比較して100倍程度高いが、冷凍機などコンプレッサ用モータに使用した場合、冷媒として用いる代替フロンと樹脂バインダが反応してしまい、耐溶媒性に問題がある。また、特開平5-121220号公報には、ボンド磁石粉をゾル・ゲル法等によりセラミックスバインダでコートし、その後に成形金型中で直接圧縮通電して、フル密度磁石を得る方法が提案されている。この方法は、磁石を硬化する際にセラミックスバインダのガラス転移温度（約500℃）以上の加熱を必要とするためにバインダとの反応等による磁粉の劣化が進行し、実用に供し得るに十分な磁気特性と高い比電気抵抗を両立させることは困難である。特開平10-321427号公報には、ボンド磁石粉を液状無機バインダを用いてより低温で結着する方法が提案されているが、実用十分な磁気特性と高比電気抵抗を具備することが困難である。特開平9-186010号及び特開平10-163055号公報には、希土類磁石粉末を絶縁物で希土類磁石と反応しないアルカリ金属又はアルカリ土類金属のフッ化物及び／又は酸化物と混合したのち密度化して、高比電気抵抗磁石を得る方法が提案されている。この方法では高い比電気抵抗を得るためには相当量の絶縁物を添加せねばならず、希土類磁石相の体積が大きく減少し、磁化が低下するため、実用十分な磁気特性と高い比電気抵抗の両立が難しい。特開平11-329809号公報では、磁化方向に電気抵抗率の高い層を積層し、表皮効果により渦電流の流れる部分の比電気抵抗だけを上げてジュール熱による発熱を抑えようという方法が提案されている。これは外部磁界の変動方向と磁石の磁化方向が平行な場合は有効だが、実際のモータのようにその方向が絶えずずれるような場合有効ではない。

【0010】従って、本発明は高い比電気抵抗と十分な磁気特性を共に備えた高比電気抵抗性希土類磁石及びその製造方法を提供することを目的とする。

【0011】

【課題を解決するための手段及び発明の実施の形態】本発明者らは、高い比電気抵抗と十分な磁気特性を併せ持つような希土類元素・鉄・ボロン系焼結磁石を得るために様々な方法を考察、検討した。その結果、R-T-B合金[RはYを含む希土類元素の1種以上（以下同じ）、TはFe及び／又はCo（以下同じ）]及び／又はR-T-M-B合金[MはAl, Si, Ti, V, Cr, Cu, Zr, Nb, Mo, Ta, W, Ga, Snのいずれかより選ばれた1又は2以上の元素（以下同じ）]のインゴット合金又は急冷薄帯を粉砕する工程において、B₂O₃粉末を混合、粉砕し、磁場中配向成形後、焼結することにより、1種以上の希土類酸化物R'
mOn [R'はYを含む希土類元素の1種以上で、m、

nは、それぞれCeの場合1, 2又は2, 3、Prの場合2, 3又は6, 11、Tbの場合2, 3又は4, 7、その他は全て2, 3である（以下同じ）]及び／又は希土類元素R' [R'はYを含む希土類元素の1種以上（以下同じ）]とBとの複合酸化物を含み、残部がR-T-B合金及び／又はR-T-M-B合金である高比電気抵抗性希土類磁石が得られることを見出し、更に諸条件を確認して本発明を完成した。

【0012】即ち、本発明は、(1) 1種以上の希土類酸化物R'
mOn及び／又は希土類元素R' とBとの複合酸化物を含み、残部がR-T-B合金及び／又はR-T-M-B合金であることを特徴とする高比電気抵抗性希土類磁石、及び、(2) R-T-B合金及び／又はR-T-M-B合金のインゴット合金又は急冷薄帯を粉砕する工程において、B₂O₃粉末を混合、粉砕し、磁場中配向成形後、焼結することにより、高比電気抵抗性希土類磁石の製造方法を提供する。

【0013】以下、本発明を詳細に説明する。本発明によれば、

①R-T-B合金及び／又はR-T-M-B合金のインゴット合金又は急冷薄帯を粉砕する工程において、B₂O₃粉末を混合、粉砕し、磁場中配向成形後、焼結する
②必要により、得られた合金を熱処理することにより、1種以上の希土類酸化物R'
mOn及び／又は希土類元素R' とBとの複合酸化物を含み、残部がR-T-B合金及び／又はR-T-M-B合金であることを特徴とする高比電気抵抗性希土類磁石を得ることができる。

【0014】ここで、RはYを含む希土類元素の1種以上であり、具体的にはY, La, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Luから選ばれる。TはFe及び／又はCo、MはAl, Si, Ti, V, Cr, Cu, Zr, Nb, Mo, Ta, W, Ga, Snより選ばれる。

【0015】R'はYを含む希土類元素の1種以上であり、具体的にはY, La, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Luから選ばれる。この場合、m, nは、それぞれCeの場合1, 2又は2, 3、Prの場合2, 3又は6, 11、Tbの場合2, 3又は4, 7であり、その他の元素の場合はいずれも2, 3である。R'はYを含む希土類元素の1種以上であり、具体的にはY, La, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Luから選ばれる。

【0016】本発明においては、上記のようにR-T-B合金及び／又はR-T-M-B合金の粉砕時にB₂O₃粉末を混合、粉砕し、これを磁場中配向成形後、焼結するものであるが、この際、混合したB₂O₃粉末が焼結処理中に、

①450℃付近でB₂O₃が溶ける、

② R-T-B 合金及び／又は R-T-M-B 合金粉末を
一様に濡らし包み込む、

③ 液体となった B_2O_3 が R-T-B 合金及び／又は R-T-M-B 合金粉末と反応し還元されて、希土類酸化物 $R'mOn$ 及び／又は希土類元素 R'' と B の複合酸化物となる、

④ 全ての還元反応が終了した段階で、希土類酸化物 $R'mOn$ 、希土類元素 R'' と B の複合酸化物はそのまま磁石中に分散状態で残る、

⑤ 残った R-T-B 合金及び／又は R-T-M-B 合金粉末、及びその合金と還元されて生じた B の合金で液相焼結が起こる

という過程を経ることによって、絶縁体である希土類酸化物 $R'mOn$ 及び／又は希土類元素 R'' と B の複合酸化物が、磁性相である R-T-B 合金及び／又は R-T-M-B 合金組成物中に均一に分散している高比電気抵抗性希土類磁石が得られる。

【0017】以下に、この磁石の製造方法を詳述する。最初に R-T-B 合金及び／又は R-T-M-B 合金組成物の粉末を作製する。この合金組成物の粉末は、例えば溶解、鑄造したインゴット合金を粉砕してもよい、酸化物等から直接還元拡散法を用いて作製してもよい。溶解したものをストリップキャスティング法を用いて急冷した急冷薄帯を粉砕したものでもよい。また、得られた合金を水素化、脱水素化することによって粉砕したものでもよい。更に、二合金法を用いて、主相の $R_2(Fe, Co)$ 14B 相を主に含む主相合金粉末と、希土類リッチ相を含む助剤合金粉末を別々に前記のように作製して、混合したものでもよい。

【0018】この R-T-B 合金及び／又は R-T-M-B 合金粉末中の R は、Y を含む希土類元素の 1 種以上で、主成分として Pr, Nd を用いることが好ましい。また、R 量全体の 0~20 重量%を Dy, Tb で置換することは保磁力の向上に効果がある。入手上の便宜などの理由から R として 2 種以上の混合物（ミッシュメタル、ジジム等）を用いてもよい。なお、R は純希土類元素でなくてもよく、工業上入手可能な範囲で製造上不可避な不純物を含有するものでも差し支えない。R は 8~40 原子%を占めることが望ましい。R がこの範囲より少ないと還元に使われる R が不足し、合金中に粗大な $\alpha-Fe$ が析出することにより保磁力を低下させるおそれがある。また、R がこの範囲より多いと強磁性相である主相の $R_2(Fe, Co)$ 14B 相の存在比が少なくなり、残留磁化を低下させるおそれがある。

【0019】R-T-B 合金及び／又は R-T-M-B 合金粉末中の T は、Fe 及び／又は Co であるが、合金中の 50~80 原子%が好ましい。T がこの範囲より少ないと、強磁性相である主相の $R_2(Fe, Co)$ 14B 相の存在比が少なくなり、残留磁化が低下するおそれがある。一方、T がこの範囲より多いと、合金中に粗大な

$\alpha-Fe$ が析出することにより保磁力が低下するおそれがある。

【0020】R-T-B 合金及び／又は R-T-M-B 合金粉末中の B は 0.1~10 原子%が好ましい。前記範囲外では、強磁性相である主相の $R_2(Fe, Co)$ 14B 相の存在比が少なくなり、残留磁化を低下させるおそれがある。

【0021】R-T-B 合金及び／又は R-T-M-B 合金粉末中の R, T, B の組成は、上記範囲内で、必要とされる保磁力及び残留磁化に応じて、決めればよい。

【0022】R-T-M-B 合金粉末中の M は、Al, Si, Ti, V, Cr, Cu, Zr, Nb, Mo, Ta, W, Ga, Sn のいずれかより選ばれた 1 又は 2 以上の元素である。これらの元素は、保磁力を増加させる効果があるが、残留磁化を低下させる。必要とされる保磁力及び残留磁化に応じて、その配合量を決めればよい。

【0023】本発明においては、上記 R-T-B 合金及び／又は R-T-M-B 合金粉末を B_2O_3 粉末と混合する。 B_2O_3 粉末の混合量は、0.0001~15 重量%、更に好ましくは 0.01~10 重量%とすることが好ましい。 B_2O_3 粉末の混合量が少なすぎると、還元されて生じる絶縁体の希土類酸化物 $R'mOn$ 及び／又は希土類元素 R'' と B の複合酸化物の量が少なくなつて、十分に焼結後の磁石の比電気抵抗を上げることができない場合がある。逆に B_2O_3 粉末の混合量が多すぎると、強磁性相である主相の $R_2(Fe, Co)$ 14B 相の存在比が少なくなり、実用上十分な磁気特性が出ない場合がある。 B_2O_3 粉末の混合量は、焼結磁石に必要な比電気抵抗と磁気特性との兼ね合いで決めればよい。

【0024】 B_2O_3 粉末の平均粒径は 0.1 μm ~5 mm が好ましい。粒径が 0.1 μm 未満では、酸化物粉末は凝集してしまつて、粒径の大きな酸化物粉末を使用した場合と同じ結果になる。一方、5 mm を超える粒径の B_2O_3 粉末を使用すると、混合時に希土類合金粉末と十分に分散した状態で混合できないおそれがある。

【0025】 B_2O_3 粉末を前記のような合金組成物粉末に混合後微粉砕し、次に磁場中成形するが、磁場中配向成形の条件は、磁場 5~20 kOe、成形圧力 300~2000 kgf/cm² が好ましい。

【0026】次いで磁場中配向成形した圧粉体を焼結する。焼結条件は、 N_2 、Ar 等の不活性雰囲気中又は真空中で、焼結温度は 1000~1200℃がよい。焼結温度が 1000℃より低いと、焼結体の密度が十分上がらず、同時に保磁力も上がらない場合がある。焼結温度が 1200℃より高くなると、 $R_2(Fe, Co)$ 14B 相の融点を超えるため、大きく粒成長し保磁力が下がるおそれがある。

【0027】焼結後、更に磁気特性を向上させるために

熱処理を行うとよい。この熱処理は、一般に、温度 400～800℃、時間 0.5～10 時間で昇温、保持、降温というパターンで行うが、必要に応じてこれを繰り返したり、段階又は連続的に温度を変化させるパターンでもよい。

【0028】本発明の焼結磁石には、原料合金の R-T-B 合金及び／又は R-T-M-B 合金の B_2O_3 粉末への還元反応により生じた、1 種以上の希土類酸化物 $R'mOn$ 及び／又は希土類元素 R'' と B との複合酸化物が含まれていて、これが磁石中に一様に分散していることにより、比電気抵抗を上げている。

【0029】上記のようにして得られた本発明の希土類磁石は、1 種以上の希土類酸化物 $R'mOn$ (R' は Y を含む希土類元素の 1 種以上で、m, n は、それぞれ Ce の場合 1, 2 又は 2, 3、Pr の場合 2, 3 又は 6, 11、Tb の場合 2, 3 又は 4, 7、その他は全て 2, 3 である) 及び／又は希土類元素 R'' (R'' は Y を含む希土類元素の 1 種以上) と B との複合酸化物を含み、残部が R-T-B 合金 (R は Y を含む希土類元素の 1 種以上、T は Fe 及び／又は Co) 及び／又は R-T-M-B 合金 (R は Y を含む希土類元素の 1 種以上、T は Fe 及び／又は Co、M は Al, Si, Ti, V, Cr, Cu, Zr, Nb, Mo, Ta, W, Ga, Sn のいずれかより選ばれた 1 又は 2 以上の元素) であることを特徴とする高比電気抵抗性希土類磁石であり、上記 R-T-B 合金及び／又は R-T-M-B 合金が、 $Nd_2Fe_{14}B$ 型 ($R_2(Fe, Co)_{14}B$) の結晶構造を持つ相を有するものであり、具体的に合金中における R が 8～20 原子%、B が 2～25 原子%、T 又は T+M が残部であることが好ましい。

【0030】更に希土類酸化物 $R'mOn$ 及び／又は希土類元素 R'' と B との複合酸化物の含有量は、合計で 0.0005～40 重量%、より好ましくは 0.5～30 重量%となるようにするのが好ましい。酸化物の量が 0.0005 重量%未満では、量が少なすぎて比電気抵抗を上げる効果を示さない場合があり、40 重量%を超えると、焼結磁石の磁気特性、特に飽和磁化を落とすおそれがある。希土類酸化物 $R'mOn$ の含有量は、焼結

磁石に必要なとされる比電気抵抗と磁気特性との兼ね合いで決めればよい。

【0031】

【実施例】以下、実施例及び比較例を示し、本発明を具体的に説明するが、本発明は下記の実施例に制限されるものではない。

【0032】【実施例 1～15】表 1 に記載の組成となるように金属原料を溶解し、合金インゴットを得た。この合金インゴットを粗粉碎した粉末に、平均粒径約 0.5 mm の B_2O_3 粉末を全体で表 2 に記載の重量%になるように様々な割合で混合し、この粉末をジェットミルを用いて微粉碎した。得られた粉末を磁場中配向成形後、Ar 雰囲気中で 1100℃で 2 時間焼結して、焼結磁石を作製した。

【0033】表 3 に得られた焼結磁石の磁気特性、酸化物の含有量、及び四端子法にて測定した比電気抵抗を記す。表 3 に示されているように、希土類酸化物の添加量の増加により、残留磁束密度は低下するが比電気抵抗が増加する。

【0034】また、得られた焼結磁石について E PMA を用い、特性 X 線分布像を観測した結果、R-T-B 合金、R-T-M-B 合金、希土類酸化物 $R'mOn$ 、希土類元素 R'' と B との複合酸化物の存在が確認され、粉末 X 線回折では R-T-B/R-T-M-B の $Nd_2Fe_{14}B$ 型の結晶構造を持つ相が確認された。

【0035】【比較例 1, 2】表 1 に記載の組成となるように金属原料を溶解し、合金インゴットを得た。 B_2O_3 粉末を加えないこと以外は、実施例 1 と同様に操作して、焼結磁石を作製した。表 3 に、この焼結磁石の磁気特性、比電気抵抗を記す。

【0036】また、得られた焼結磁石について E PMA を用い、特性 X 線分布像を観測した結果、R-T-B 合金、R-T-M-B 合金の存在のみが確認され、粉末 X 線回折では R-T-B/R-T-M-B の $Nd_2Fe_{14}B$ 型の結晶構造を持つ相が確認された。

【0037】

【表 1】

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		Nd	Dy	Tb	Fe	Co	B	Al	Cu	Zr	V	Cr	Si	Ti	Nb	Mo	Ta	W	Ga	Sn
実施例	1	16	-	-	75	2	7	-	-	-	-	-	-	-	-	-	-	-	-	-
	2	18.5	-	-	73.5	2	6	-	-	-	-	-	-	-	-	-	-	-	-	-
	3	22	-	-	73.5	1.5	3	-	-	-	-	-	-	-	-	-	-	-	-	-
	4	30	-	-	67	1.5	1.5	-	-	-	-	-	-	-	-	-	-	-	-	-
	5	16.5	1	1	71.5	2	6	0.5	0.5	1	-	-	-	-	-	-	-	-	-	-
	6	16.5	1	1	71.5	2	6	0.5	0.5	0.5	0.5	-	-	-	-	-	-	-	-	-
	7	16.5	1	1	72	2	6	0.5	0.2	0.3	0.3	0.2	-	-	-	-	-	-	-	-
	8	16.5	1	1	72	2	6	0.5	0.2	0.2	0.2	0.2	0.2	-	-	-	-	-	-	-
	9	16.5	1	1	72	2	6	0.5	0.2	0.2	0.2	0.2	0.1	0.1	-	-	-	-	-	-
	10	16.5	1	1	71.5	2	6	0.5	0.2	0.3	0.3	0.2	0.2	0.2	0.1	-	-	-	-	-
	11	16.5	1	1	71.5	2	6	0.5	0.2	0.3	0.2	0.2	0.2	0.2	0.1	0.1	-	-	-	-
	12	16.5	1	1	71	2	6	0.5	0.2	0.3	0.3	0.2	0.2	0.2	0.2	0.2	0.2	-	-	-
	13	16.5	1	1	71	2	6	0.5	0.2	0.3	0.3	0.2	0.2	0.2	0.2	0.2	0.1	0.1	-	-
	14	16.5	1	1	71	2	6	0.5	0.2	0.3	0.2	0.2	0.2	0.2	0.2	0.2	0.1	0.1	0.1	-
	15	16.5	1	1	71	2	6	0.4	0.2	0.3	0.2	0.2	0.2	0.2	0.2	0.2	0.1	0.1	0.1	0.1
比較例	1	15	-	-	75	2	8	-	-	-	-	-	-	-	-	-	-	-	-	-
	2	13	1	1	72.5	2	8	0.4	0.2	0.3	0.2	0.2	0.2	0.2	0.2	0.2	0.1	0.1	0.1	0.1

単位は原子%

【0039】

【0038】

【表3】

【表2】

		B ₂ O ₃ 混合量 [重量%]
実施例	1	0.5
	2	1.5
	3	3
	4	6
	5	1.5
	6	1.5
	7	1.5
	8	1.5
	9	1.5
	10	1.5
	11	1.5
	12	1.5
	13	1.5
	14	1.5
	15	1.5
比較例	1	-
	2	-

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		酸化物含有量 [重量%]	保磁力 [kOe]	残留磁化 [kG]	比電気抵抗 [Ωcm]
実施例	1	3.2	13.8	11.0	3.9×10^{-4}
	2	6.6	10.6	10.5	8.3×10^{-4}
	3	13.5	7.8	9.5	1.8×10^{-3}
	4	27.8	4.0	8.0	7.4×10^{-3}
	5	6.7	12.5	10.0	8.7×10^{-4}
	6	6.6	12.3	9.9	8.6×10^{-4}
	7	6.6	12.3	9.7	8.4×10^{-4}
	8	6.5	12.2	9.6	8.5×10^{-4}
	9	6.8	12.0	9.5	8.4×10^{-4}
	10	6.6	12.1	9.5	8.7×10^{-4}
	11	6.8	12.4	9.4	8.6×10^{-4}
	12	6.5	12.3	9.3	8.5×10^{-4}
	13	6.6	12.3	9.3	8.6×10^{-4}
	14	6.7	12.2	9.2	8.4×10^{-4}
	15	6.6	12.4	9.2	8.5×10^{-4}
比較例	1	—	13.5	12.0	1.8×10^{-4}
	2	—	15.6	11.7	1.9×10^{-4}

【0040】

【発明の効果】本発明によれば、高い保磁力及びモータ
等変動する磁界にさらされるような使用条件でも渦電流

の発生が抑えられる大きな比電気抵抗を持つ焼結磁石
を、低コストで製造できる。

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MINOWA TAKEHISA

(54) HIGH-RESISTIVITY RARE EARTH MAGNET AND ITS MANUFACTURING METHOD

(57)Abstract:

PROBLEM TO BE SOLVED: To provide a sintered magnet which has a high coercive force, is high enough in resistivity to restrain generation of an eddy current even if it is used for a motor where it is exposed to a fluctuating magnetic field, and manufactured at a low cost.

SOLUTION: This high resistivity rare earth magnet is formed of material that contains one or more rare earth oxides $R'mOn$ (wherein, R' is one or more rare earth elements including Y; (m) and (n) are 1, 2 or 2, 3 when R' is Ce, 2, 3 or 6, 11 when R' is Pr, 2, 3 or 4, 7 when R' is Tb, and 2, 3 when R' is other rare earth elements) and/or rare earth element R'' (R'' is one or more earth elements, including Y), a B composite oxide, and the residue which is R-T-B alloy (wherein, R is one or more rare earth elements including Y; T is Fe and/or Co) and/or R-T-M-B alloy (wherein, R is one or more earth elements including Y; T is Fe and/or Co; M is one or more elements selected out of Al, Si, Ti, V, Cr, Cu, Zr, Nb, Mo, Ta, W, Ga, and Sn).

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CLAIMS

[Claim(s)]

[Claim 1] One or more sorts of rare earth oxide $R'mOn$ (R' is one or more sorts of the rare earth elements containing Y m and n) In the case of 1, 2 or 2 and 3, and Pr, in the case of 2, 3 or 6 and 11, and Tb, in Ce, and/or all of 2, 3 or 4 and 7, and others are 2 and 3, they contain the multiple oxide of rare-earth-elements R'' (one or more sorts of the rare earth elements in which R'' contains Y), and B, respectively. The remainder is a R-T-B alloy (one or more sorts of the rare earth

elements in which R contains Y, and T are Fe and/or Co), and/or a R-T-M-B alloy (the rare earth elements containing Y one or more sorts of R). It is the Taka specific resistance nature rare earth magnet characterized by being 1 or two or more elements with which T was chosen from Co and Fe and/or M were chosen from aluminum, Si, Ti, V, Cr, Cu, Zr, Nb, Mo, Ta, W, Ga, or Sn.

[Claim 2] The Taka specific resistance nature rare earth magnet according to claim 1 characterized by having the phase in which R-T-B and/or a R-T-M-B alloy have the crystal structure of a Nd₂Fe₁₄B mold.

[Claim 3] The Taka specific resistance nature rare earth magnet according to claim 1 or 2 whose content of the multiple oxide of rare earth oxide R'mOn and/or R", and B is 0.0005 - 40 % of the weight.

[Claim 4] A R-T-B alloy (one or more sorts of the rare earth elements in which R contains Y, and T are Fe and/or Co), and/or a R-T-M-B alloy (the rare earth elements containing Y one or more sorts of R) In the process which grinds the ingot alloy or quenching thin band of 1 as which T was chosen as from Co and Fe and/or M were chosen from aluminum, Si, Ti, V, Cr, Cu, Zr, Nb, Mo, Ta, W, Ga, or Sn, or 2 or more elements The manufacture approach of the Taka specific resistance nature rare earth magnet which mixes and grinds B-2O₃ powder and is characterized by sintering after orientation shaping in a magnetic field.

[Claim 5] The manufacture approach of the Taka specific resistance nature rare earth magnet according to claim 4 that eight to 40 atom % and B are [a R-T-B alloy / 0.1 to 10 atom % and the remainder] T for R.

[Claim 6] The manufacture approach of the Taka specific resistance nature rare earth magnet according to claim 4 or 5 which makes the amount of mixing of B-2O₃ powder 0.0001 - 15 % of the weight.

[Claim 7] The manufacture approach of claim 4 characterized by the mean particle diameter of B-2O₃ powder being 0.1 micrometers - 5mm thru/or the Taka specific resistance nature rare earth magnet of six given in any 1 term.

[Claim 8] The manufacture approach of the Taka specific resistance nature rare earth magnet characterized by heat-treating the Taka specific resistance nature

rare earth magnet manufactured by the approach of seven claim 4 thru/or given in any 1 term.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to a useful high specific resistance nature rare earth magnet and its manufacture approach especially in the industrial field of a rotating equipment, electronic parts, and an electrical machinery and apparatus.

[0002]

[Description of the Prior Art] The permanent magnet which consists of R (R is the same one or more sorts of the rare earth elements containing Y and the following), Fe, Co, and B, and the rare earth magnet which uses Nd as a principal component especially as R are widely used in the field of an electron and electrical machinery and apparatus industry from the height of the magnetic properties.

[0003] For example, the conventionally cheap ferrite magnet has mainly been

used for a permanent magnet type rotating equipment. However, to the demand of the further miniaturization of a rotating equipment, and increase in efficiency, although it was expensive, the high rare earth magnet of magnetic properties came to be used in recent years. Among the rare earth magnets generally marketed, since a Sm-Co system magnet has a high Curie temperature, its temperature change of magnetic properties is small. Moreover, corrosion resistance is also high and does not need surface treatment. However, since Co is used as a raw material, it is very expensive. On the other hand, a Nd-Fe-B system magnet has the highest saturation magnetization in a permanent magnet, and since Co is not used from a raw material, it is cheap. However, since Curie temperature is low, the temperature change of magnetic properties is large and inferior to thermal resistance. Since corrosion resistance is also inferior to coincidence, suitable surface treatment is needed depending on an application.

[0004]

[Problem(s) to be Solved by the Invention] Since a rare earth magnet is a metal, specific resistance is as small as about $1/100$ 100-200microomegacm grade of the specific resistance of the ferrite which is an oxide. Therefore, when used being exposed to the field to which this rare earth magnet is changed by rotating equipments, such as a motor, the eddy current generated by electromagnetic induction flows in large quantities, and a permanent magnet generates heat with the Joule's heat by that current. If the temperature of a permanent magnet becomes high, since especially a Nd-Fe-B system magnet has the large temperature change of magnetic properties, magnetic properties will fall and, as a result, the effectiveness of a motor will also fall.

[0005] Enlarging magnetic coercive force is mentioned as one of the cure of this. By enlarging magnetic coercive force, magnetic thermal resistance lifting-comes to be hard of increase and irreversible demagnetization. Especially a Nd-Fe-B system magnet has the large temperature change of the lowness of the Curie temperature to magnetic properties, and various attempts which heighten the coercive force have been made.

[0006] The approach by which effectiveness showed up most although the coercive force is heightened was the way of permuting a part of R of the R₂ (Fe, Co) 14 B phase used as the main phase by heavy rare earth, such as Dy and Tb, and raising a crystal magnetic anisotropy. However, magnetic saturation magnetization becomes small, and since heavy rare earth is very expensive, this approach goes up [magnetic cost] it.

[0007] There is the approach of dividing a permanent magnet into the approach for suppressing an eddy current small, and insulating to it. Although this approach has the effectiveness which makes calorific value small in inverse proportion to the magnetic number of partitions, it will raise the cost of magnet processing.

[0008] Then, the attempt which is going to enlarge specific resistance of the rare earth magnet itself is made as a radical cure to an eddy current.

[0009] Although the specific resistance of the Nd-Fe-B system rare earth bond magnet which used the resin binder is high about 100 times as compared with a Nd-Fe-B system sintered magnet, when it is used for motors for compressors, such as a refrigerator, the chlorofluorocarbon-replacing material and resin binder which are used as a refrigerant react, and a problem is in solvent-proof nature. Moreover, the coat of the bond magnet powder is carried out with a ceramic binder with a sol-gel method etc., direct compression energization is carried out in shaping metal mold after that, and the method of obtaining a full consistency magnet is proposed by JP,5-121220,A. This approach is difficult to reconcile sufficient magnetic properties to be able to present [for degradation of the magnetic powder by a reaction with a binder etc. to advance, since heating more than the glass transition temperature (about 500 degrees C) of a ceramic binder is needed in case a magnet is hardened, and] practical use, and high specific resistance. although the approach of binding bond magnet powder with low temperature more using a liquefied inorganic binder is proposed by JP,10-321427,A -- practical use -- it is difficult to provide sufficient magnetic properties and high specific resistance. An after consistency is formed and the approach of

obtaining a high specific resistance magnet for which rare earth magnet powder was mixed with the fluoride of a rare earth magnet, the alkali metal which does not react, or alkaline earth metal, and/or the oxide with the insulating material is proposed by JP,9-186010,A and JP,10-163055,A. since the insulating material of a considerable amount must be added in order to obtain high specific resistance by this approach, the volume of a rare earth magnet phase decreases greatly and magnetization falls -- practical use -- coexistence of sufficient magnetic properties and high specific resistance is difficult. The laminating of the high layer of electrical resistivity is carried out in the magnetization direction, only the specific resistance of the part into which an eddy current flows according to a skin effect is raised in JP,11-329809,A, and the approach of suppressing generation of heat by the Joule's heat is proposed. It is not effective when [effective when the fluctuation direction of an external magnetic field and the magnetic magnetization direction are parallel as for this] the direction shifts continuously like an actual motor but.

[0010] Therefore, this invention aims at offering the high specific resistance nature rare earth magnet equipped with both high specific resistance and sufficient magnetic properties, and its manufacture approach.

[0011]

[The means for solving a technical problem and the gestalt of implementation of invention] this invention persons considered and examined various approaches, in order to obtain the rare earth elements, the iron, and a boron system sintered magnet it has a sintered magnet and high specific resistance and sufficient magnetic properties. Consequently, one or more (it is below the same) sorts of the rare earth elements in which R-T-B alloy [R contains Y, T the ingot alloy or quenching thin band of Fe, Co(it is below the same)], and/or a R-T-M-B alloy [1 or 2 or more elements (it is below the same) with which M was chosen from aluminum, Si, Ti, V, Cr, Cu, Zr, Nb, Mo, Ta, W, Ga, or Sn] One or more sorts of rare earth oxide $R'mOn$ [R' is one or more sorts of the rare earth elements containing Y by setting at the process to pulverize, mixing and grinding B-2O₃

powder, and sintering after orientation shaping in a magnetic field. m and n respectively -- Ce -- a case -- one -- two -- or -- two -- three -- Pr -- a case -- two -- three -- or -- six -- 11 -- Tb -- a case -- two -- three -- or -- four -- seven -- others -- all -- two -- three -- it is (below the same) --] -- and/or -- rare earth elements -- R -- ' -- ' -- [-- R -- ' -- ' -- Y -- containing -- rare earth elements -- one -- a sort -- more than (below the same) --] -- B -- a multiple oxide -- containing -- It found out that the high specific resistance nature rare earth magnet whose remainders are a R-T-B alloy and/or a R-T-M-B alloy was obtained, terms and conditions were checked further, and this invention was completed.

[0012] Namely, this invention contains the multiple oxide of rare earth oxide R'mOn beyond (1) 1 sort and/or rare-earth-elements R", and B. In the process which grinds the ingot alloy or quenching thin band of the high specific resistance nature rare earth magnet characterized by the remainders being a R-T-B alloy and/or a R-T-M-B alloy and (2) R-T-B alloy, and/or a R-T-M-B alloy B-2O₃ powder is mixed and ground and the manufacture approach of the high specific resistance nature rare earth magnet characterized by sintering after orientation shaping in a magnetic field is offered.

[0013] Hereafter, this invention is explained to a detail. In the process which grinds the ingot alloy or quenching thin band of **R-T-B alloy and/or a R-T-M-B alloy according to this invention Mix and grind B-2O₃ powder and after orientation shaping in a magnetic field according to ** need to sinter By heat-treating the obtained alloy, the high specific resistance nature rare earth magnet characterized by the remainders being a R-T-B alloy and/or a R-T-M-B alloy can be obtained including the multiple oxide of one or more sorts of rare earth oxide R'mOn and/or rare-earth-elements R", and B.

[0014] Here, R is one or more sorts of the rare earth elements containing Y, and is specifically chosen from Y, La, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, and Lu. T is chosen from Co and Fe and/or M are chosen from aluminum, Si, Ti, V, Cr, Cu, Zr, Nb, Mo, Ta, W, Ga, and Sn.

[0015] R' is one or more sorts of the rare earth elements containing Y, and is

specifically chosen from Y, La, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, and Lu. In this case, in Ce, in the case of 1, 2 or 2 and 3, and Pr, m and n are 2, 3, or 4 and 7, respectively in the case of 2, 3 or 6 and 11, and Tb, and, in the case of other elements, it is all 2 and 3. R" is one or more sorts of the rare earth elements containing Y, and is specifically chosen from Y, La, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, and Lu.

[0016] Although it sets to this invention, B-2O₃ powder is mixed and ground as mentioned above at the time of grinding of a R-T-B alloy and/or a R-T-M-B alloy and this is sintered after orientation shaping in a magnetic field Under the present circumstances, B-2O₃ mixed powder melts [B-2 O₃] near **450 degree C during sintering processing. ** Wet uniformly a R-T-B alloy and/or R-T-M-B alloy powder, and wrap them in. ** ** which B-2 O₃ used as a liquid reacts with a R-T-B alloy and/or R-T-M-B alloy powder, is returned, and becomes rare earth oxide R'mOn and/or rare-earth-elements R", and the multiple oxide of B -- in the phase which all reduction reactions ended The multiple oxide of rare earth oxide R'mOn, rare-earth-elements R", and B remains in the state of distribution into a magnet as it is. ** By passing through the process in which liquid phase sintering happens with the alloy of B which it was returned with the remaining R-T-B alloy and/or R-T-M-B alloy powder, and its alloy, and was produced The high specific resistance nature rare earth magnet which the multiple oxide of rare earth oxide R'mOn and/or rare-earth-elements R" which are an insulator, and B is distributing to homogeneity in the R-T-B alloy which is a magnetic phase, and/or a R-T-M-B alloy constituent is obtained.

[0017] Below, the manufacture approach of this magnet is explained in full detail. The powder of a R-T-B alloy and/or a R-T-M-B alloy constituent is produced first. The powder of this alloy constituent may grind the ingot alloy dissolved and cast, for example, and may produce it using a direct reduction diffusion method from an oxide etc. What ground the quenching thin band which quenched what was dissolved using the strip casting method may be used. Moreover, what ground the obtained alloy hydrogenation and by carrying out dehydrogenation may be

used. Furthermore, what produced separately the main phase alloy powder which mainly contains the R₂ (Fe, Co) 14 B phase of the main phase, and the assistant alloy powder containing a rare earth rich phase as mentioned above, and was mixed using two alloying methods may be used.

[0018] R in this R-T-B alloy and/or R-T-M-B alloy powder is one or more sorts of the rare earth elements containing Y, and it is desirable to use Pr and Nd as a principal component. Moreover, permuting 0 - 20% of the weight of the whole amount of R by Dy and Tb has effectiveness in improvement in coercive force. Since [any] it is [on acquisition] expedient, two or more sorts of mixture (a misch metal, didym, etc.) may be used as R. In addition, R may not be pure rare earth elements and what contains an unescapable impurity on manufacture in the available range on industry does not interfere. As for R, it is desirable to occupy 8 - 40 atom %. If there is less R than this range, R used for reduction runs short, and when big and rough alpha-Fe deposits in an alloy, there is a possibility of reducing coercive force. Moreover, if there is more R than this range, the abundance ratio of the R₂ (Fe, Co) 14 B phase of the main phase which is a ferromagnetic phase will decrease, and there is a possibility of reducing residual magnetization.

[0019] Although T in a R-T-B alloy and/or R-T-M-B alloy powder is Fe and/or Co, 50 in an alloy - its 80 atom % are desirable. When there is less T than this range, the abundance ratio of the R₂ (Fe, Co) 14 B phase of the main phase which is a ferromagnetic phase decreases, and there is a possibility that residual magnetization may fall. On the other hand, when there is more T than this range, there is a possibility that coercive force may decline into an alloy when big and rough alpha-Fe deposits.

[0020] B in a R-T-B alloy and/or R-T-M-B alloy powder has desirable 0.1 - 10 atom %. Said abundance ratio of the R₂ (Fe, Co) 14 B phase of the main phase which is a ferromagnetic phase if out of range decreases, and there is a possibility of reducing residual magnetization.

[0021] What is necessary is for the presentation of R, T, and B in a R-T-B alloy

and/or R-T-M-B alloy powder to be [above-mentioned] within the limits, and just to decide it according to the coercive force and residual magnetization which are needed.

[0022] M in R-T-M-B alloy powder is 1 or two or more elements which were chosen from aluminum, Si, Ti, V, Cr, Cu, Zr, Nb, Mo, Ta, W, Ga, or Sn. Although these elements are effective in making coercive force increase, they reduce residual magnetization. What is necessary is just to decide the loadings according to the coercive force and residual magnetization which are needed.

[0023] In this invention, the above-mentioned R-T-B alloy and/or R-T-M-B alloy powder are mixed with B-2O₃ powder. As for the amount of mixing of B-2O₃ powder, it is still more preferably desirable to consider as 0.01 - 10 % of the weight 0.0001 to 15% of the weight. If there are too few amounts of mixing of B-2O₃ powder, the amount of the multiple oxide of rare earth oxide R'mOn of an insulator and/or rare-earth-elements R" which it is returned and are produced, and B decreases, and specific resistance of the magnet after sintering may fully be unable to be raised. Conversely, if there are too many amounts of mixing of B-2O₃ powder, the abundance ratio of the R₂ (Fe, Co) 14 B phase of the main phase which is a ferromagnetic phase decreases, and practically sufficient magnetic properties may not come out. What is necessary is just to decide the amount of mixing of B-2O₃ powder on the balance of the specific resistance and magnetic properties which are needed for a sintered magnet.

[0024] The mean particle diameter of B-2O₃ powder has 0.1 micrometers - 5 desirablenm. The result as the case where condensed oxide powder in less than 0.1 micrometers, and oxide powder with a big particle size is used with the same particle size is brought. On the other hand, when B-2O₃ powder of the particle size exceeding 5mm is used, there is a possibility that it may be unmixable in the condition of having fully distributed with rare earth alloy powder, at the time of mixing.

[0025] Although B-2O₃ powder is pulverized after mixing to the above alloy constituent powder and then is fabricated among a magnetic field, the conditions

of orientation shaping in a magnetic field have a magnetic field 5 - 20kOe, and desirable compacting pressure 300 - 2000 kgf/cm².

[0026] Subsequently, the green compact which carried out orientation shaping in a magnetic field is sintered. Sintering temperature has [sintering conditions] good 1000-1200 degrees C in inert atmospheres, such as N₂ and Ar, or a vacuum. If sintering temperature is lower than 1000 degrees C, the consistency of a sintered compact may not go up enough and coercive force may not go up to coincidence, either. If sintering temperature becomes higher than 1200 degrees C, in order to exceed the melting point of R₂ (Fe, Co) 14 B phase, there is a possibility that grain growth may be carried out greatly and coercive force may fall.

[0027] It is good to heat-treat after sintering, in order to raise magnetic properties further. Although a pattern called a temperature up, maintenance, and a temperature fall generally performs this heat treatment in temperature [of 400-800 degrees C], and time amount 0.5 - 10 hours, if needed, this may be repeated or a phase or the pattern to which temperature is changed continuously is sufficient.

[0028] The multiple oxide of one or more sorts of rare earth oxide R'_mO_n and/or rare-earth-elements R'' which were produced by the reduction reaction to B-2O₃ powder of the R-T-B alloy of a raw material alloy and/or a R-T-M-B alloy, and B is contained in the sintered magnet of this invention, and specific resistance is raised when this is distributing uniformly in a magnet.

[0029] The rare earth magnet of this invention obtained as mentioned above One or more sorts of rare earth oxide R'_mO_n (R' is one or more sorts of the rare earth elements containing Y m and n) In the case of 1, 2 or 2 and 3, and Pr, in the case of 2, 3 or 6 and 11, and Tb, in Ce, and/or all of 2, 3 or 4 and 7, and others are 2 and 3, they contain the multiple oxide of rare-earth-elements R'' (one or more sorts of the rare earth elements in which R'' contains Y), and B, respectively. The remainder is a R-T-B alloy (one or more sorts of the rare earth elements in which R contains Y, and T are Fe and/or Co), and/or a R-T-M-B alloy (the rare earth

elements containing Y one or more sorts of R). It is the high specific resistance nature rare earth magnet characterized by being 1 or two or more elements with which T was chosen from Co and Fe and/or M were chosen from aluminum, Si, Ti, V, Cr, Cu, Zr, Nb, Mo, Ta, W, Ga, or Sn. It has the phase in which the above-mentioned R-T-B alloy and/or a R-T-M-B alloy have the crystal structure of a Nd₂Fe₁₄B mold (R₂(Fe, Co) ₁₄B), and it is desirable that eight to 20 atom % and B is [two to 25 atom %, T, or T+M] the remainder concretely for R in an alloy.

[0030] Furthermore, as for the content of the multiple oxide of rare earth oxide R'mOn and/or rare-earth-elements R", and B, it is desirable to make it become 0.5 - 30 % of the weight more preferably 0.0005 to 40% of the weight in total. When the amount of an oxide may not show the effectiveness that there are too few amounts and they raise specific resistance at less than 0.0005 % of the weight and exceeds 40 % of the weight, there are the magnetic properties of a sintered magnet, especially a possibility of dropping saturation magnetization. What is necessary is just to decide the content of rare earth oxide R'mOn on the balance of the specific resistance and magnetic properties which are needed for a sintered magnet.

[0031]

[Example] Although an example and the example of a comparison are shown and this invention is explained concretely hereafter, this invention is not restricted to the following example.

[0032] The metal raw material was dissolved so that it might become a presentation given in the [examples 1-15] table 1, and the alloy ingot was obtained. To the powder which carried out coarse grinding of this alloy ingot, it mixed at a various rate so that it might become weight % given [B-2O₃ powder with a mean particle diameter of about 0.5mm] in Table 2 on the whole, and to it, this powder was pulverized using the jet mill. The obtained powder was sintered at 1100 degrees C in Ar ambient atmosphere after orientation shaping in a magnetic field for 2 hours, and the sintered magnet was produced.

[0033] The magnetic properties of the sintered magnet obtained in Table 3, the content of an oxide, and the specific resistance measured with the four probe method are described. Although a residual magnetic flux density falls, specific resistance increases it by the increment in the addition of a rare earth oxide, as shown in Table 3.

[0034] Moreover, as a result of observing a characteristic-X-ray distribution image using EPMA about the obtained sintered magnet, existence of the multiple oxide of a R-T-B alloy, a R-T-M-B alloy, rare earth oxide R'mOn, rare-earth-elements R'', and B was checked, and the phase with the crystal structure of the Nd₂Fe₁₄B mold of R-T-B/R-T-M-B was checked in the powder X diffraction.

[0035] The metal raw material was dissolved so that it might become a presentation given in the [examples 1 and 2 of comparison] table 1, and the alloy ingot was obtained. Except not adding B₂O₃ powder, it was operated like the example 1 and the sintered magnet was produced. The magnetic properties of this sintered magnet and specific resistance are described in Table 3.

[0036] Moreover, as a result of observing a characteristic-X-ray distribution image using EPMA about the obtained sintered magnet, only existence of a R-T-B alloy and a R-T-M-B alloy was checked, and the phase with the crystal structure of the Nd₂Fe₁₄B mold of R-T-B/R-T-M-B was checked in the powder X diffraction.

[0037]

[Table 1]

		Nd	Dy	Tb	Fe	Co	B	Al	Cu	Zr	V	Cr	Si	Ti	Nb	Mo	Ta	W	Ga	Sn
実施例	1	16	—	—	75	2	7	—	—	—	—	—	—	—	—	—	—	—	—	—
	2	18.5	—	—	73.5	2	6	—	—	—	—	—	—	—	—	—	—	—	—	—
	3	22	—	—	73.5	1.5	3	—	—	—	—	—	—	—	—	—	—	—	—	—
	4	30	—	—	67	1.5	1.5	—	—	—	—	—	—	—	—	—	—	—	—	—
	5	16.5	1	1	71.5	2	6	0.5	0.5	1	—	—	—	—	—	—	—	—	—	—
	6	16.5	1	1	71.5	2	6	0.5	0.5	0.5	0.5	—	—	—	—	—	—	—	—	—
	7	16.5	1	1	72	2	6	0.5	0.2	0.3	0.3	0.2	—	—	—	—	—	—	—	—
	8	16.5	1	1	72	2	6	0.5	0.2	0.2	0.2	0.2	0.2	—	—	—	—	—	—	—
	9	16.5	1	1	72	2	6	0.5	0.2	0.2	0.2	0.2	0.1	0.1	—	—	—	—	—	—
	10	16.5	1	1	71.5	2	6	0.5	0.2	0.3	0.3	0.2	0.2	0.2	0.1	—	—	—	—	—
	11	16.5	1	1	71.5	2	6	0.5	0.2	0.3	0.2	0.2	0.2	0.2	0.1	0.1	—	—	—	—
	12	16.5	1	1	71	2	6	0.5	0.2	0.3	0.3	0.2	0.2	0.2	0.2	0.2	0.2	—	—	—
	13	16.5	1	1	71	2	6	0.5	0.2	0.3	0.3	0.2	0.2	0.2	0.2	0.2	0.1	0.1	—	—
	14	16.5	1	1	71	2	6	0.5	0.2	0.3	0.2	0.2	0.2	0.2	0.2	0.2	0.1	0.1	0.1	—
	15	16.5	1	1	71	2	6	0.4	0.2	0.3	0.2	0.2	0.2	0.2	0.2	0.2	0.1	0.1	0.1	0.1
比較例	1	15	—	—	75	2	8	—	—	—	—	—	—	—	—	—	—	—	—	—
	2	13	1	1	72.5	2	8	0.4	0.2	0.3	0.2	0.2	0.2	0.2	0.2	0.2	0.1	0.1	0.1	0.1

A unit is atomic % [0038].

[Table 2]

		B ₂ O ₃ 混合量 [重量%]
実施例	1	0.5
	2	1.5
	3	3
	4	6
	5	1.5
	6	1.5
	7	1.5
	8	1.5
	9	1.5
	10	1.5
	11	1.5
	12	1.5
	13	1.5
	14	1.5
	15	1.5
比較例	1	—
	2	—

[0039]

[Table 3]

		酸化物含有量 [重量%]	保磁力 [kOe]	残留磁化 [kG]	比電気抵抗 [Ωcm]
実施例	1	3.2	13.8	11.0	3.9×10^{-4}
	2	6.6	10.6	10.5	8.3×10^{-4}
	3	13.5	7.8	9.5	1.8×10^{-3}
	4	27.8	4.0	8.0	7.4×10^{-3}
	5	6.7	12.5	10.0	8.7×10^{-4}
	6	6.6	12.3	9.9	8.6×10^{-4}
	7	6.6	12.3	9.7	8.4×10^{-4}
	8	6.5	12.2	9.6	8.5×10^{-4}
	9	6.8	12.0	9.5	8.4×10^{-4}
	10	6.6	12.1	9.5	8.7×10^{-4}
	11	6.8	12.4	9.4	8.6×10^{-4}
	12	6.5	12.3	9.3	8.5×10^{-4}
	13	6.6	12.3	9.3	8.6×10^{-4}
	14	6.7	12.2	9.2	8.4×10^{-4}
	15	6.6	12.4	9.2	8.5×10^{-4}
比較例	1	—	13.5	12.0	1.8×10^{-4}
	2	—	15.6	11.7	1.9×10^{-4}

[0040]

[Effect of the Invention] According to this invention, a sintered magnet with the big specific resistance by which generating of an eddy current is suppressed also on a service condition which is exposed to fields to change, such as high coercive force, a high motor, etc., can be manufactured by low cost.

[Translation done.]